



FABRIQUES DE TABAC REUNIES SA.

RESEARCH AND DEVELOPMENT

## MONTHLY REPORT

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FEBRUARY 1982

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## I. RESEARCH

1. Special Events
2. Biotechnology
3. Chemical and Analytical Services
4. Special Projects

## II. PROCESS DEVELOPMENT

5. Nitrate Reduction
6. Pilot Plant Operations
7. Reconstituted Tobacco
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## III. PRODUCT DEVELOPMENT AND QUALITY ASSURANCE

10. Cigarette Development
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15. Material Testing
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### Key to Distribution:

- A. Complete Report
- B. All except Research Report
- C. All except Special Projects Research and those reports which might interfere with patent considerations
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PROJECT TITLE : PROTAGORAS  
PERIOD COVERED : JANUARY - FEBRUARY 1982  
WRITTEN BY : Bindler-G.-N. (GNB)

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Mexican Burley strips were partially deproteinated and analysed. The resulting tobacco was cut and cigarettes were made with it and analysed.

#### TOBACCO EXTRACTION

Mexican Burley strips were extracted with water, an acid base and an enzyme solution. The solution of the water step and the enzyme step were mixed together and partially deproteinated. The extraction conditions and the amount of the extracted protein as a % of the original protein content are shown in the table below.

	pH	Liquid	Temp °C	Time min.	Protein Extracted %
Step I	5.7	H <sub>2</sub> O + Acid	60	120	23
Step IIa	1.9	H <sub>2</sub> O + Acid-Base	60	120	19.5
Step IIb	12.5				
Step III	8.5	H <sub>2</sub> O + 0,16 mg/ml Pronase	40	240	18

The amount of protein extracted on the liquid phase was not as high as expected. The resulting tobacco was dried and stored.

#### PARTIAL DEPROTEINISATION OF THE LIQUID PHASE

The liquids at steps I and III were mixed together and with an inoculum of *Candida utilis* based on the LEAR process. The pH was set at 5.5 and the temperature at 30°. After an incubation of 12 hours, 50% of the "proteins" had been eliminated. The resulting solution was concentrated and respread on the cut tobacco.

# CIGARETTES

Cigarettes were manufactured and analysed.

	Mexican Burley	Extracted Max. Burley	Extracted Max. Burley and re- sprayed.
TA %	0.35	0.001	0.15
N-NO <sub>3</sub> %	0.28	0.000	0.000
N-NH <sub>3</sub> %	0.37	0.03	0.03
Protein %	16.05	8.05	9.00
CO MS mg/tob.	19.09	19.07	19.00
CO SS mg/tob.	63.01	76.04	74.00
NO MS mg/tob.	0.36	0.03	0.03
Aldehyde mg/tob.	140.00	178.00	176.00
HCN µg/tob.	167.00	150.00	157.00

It should be noted that the HCN did not change as much as expected (Monthly Report, Schulthess-D., PROTAGORAS, December 80). Further trials will be performed to investigate this phenomenon.

*GRZ*

GNB/jig/MARCH 3, 1982

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0000143821

PROJECT TITLE : SAVOURY  
PERIOD COVERED : JANUARY - FEBRUARY 1982  
WRITTEN BY : Ghiste-P. (PAG)

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The purpose of project SAVOURY is to prepare flavours which, when pyrolyzed with sheet or tobacco, give Burley-type flavour characteristics.

#### DETERMINATION OF FREE AMINO ACIDS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

At the beginning of January we received an HPLC 1082B. After a training period, a method for rapid amino acid quantitation was worked out. The separation was performed on a 5- $\mu$ m Tichrosorb NH<sub>2</sub> column with an acetonitrile/phosphate buffer gradient (1). The samples were injected without undergoing a derivatization procedure and were separated within 30 minutes.

#### SERVICE FOR OTHER GROUPS

Upon the request of Product Development, cooked flavour type P-15/1 was produced for project COBRA.

Cooked flavour P-15/1 (2) appeared very promising as it "darkened" the taste of reconstituted tobacco to make it more like air-cured tobacco. It was also uniform and its intensity constant. It had the disadvantage however of having a slightly fruity after taste.

#### REFERENCES

- (1) Rainer Schuster, Hewlett-Packard GmbH, 7517 Waldbrom, West Germany.
- (2) Ghiste-P., Monthly Report, May 1981.

PAG/jig/MARCH 4, 1982



0000143822

PROJECT TITLE : HEAT  
PERIOD COVERED : JANUARY 15 - FEBRUARY 15, 1982  
WRITTEN BY : Piadé-J.-J. (JJP)

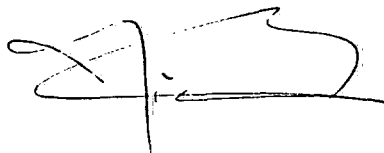
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The aim of project HEAT is to improve the physical and organoleptic properties of tobacco by heat treatment (1).

Three types of Burley tobacco were selected for heat treatment studies. Preliminary experiments have been initiated using airtight cylinders provided by PM Richmond.

#### REFERENCE

(1) Piadé-J.-J., PME Monthly Progress Report, January 1982 p.2.



JJP/jig/March 2, 1982

0000143823

PROJECT TITLE : SALAMANDER II  
PERIOD COVERED : JANUARY 18 - FEBRUARY 19, 1982  
WRITTEN BY : Murray-M. (MUM)

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The separation obtained between cysteine and cystine on a Partisil SAX anion exchange column was found to be insufficient. The use of a polar reverse phase column, Spherisorb CN, gave reasonable cysteine/cystine separation particularly when used in an ion pair mode with sodium dodecyl sulfate as counter ion. Analysis of the  $\text{NO}_2$ /cysteine reaction solution prepared under conditions used for ISH measurement gave a larger cysteine peak after reaction, together with the formation of cystine and a number of other peaks. This confirms the results obtained with a Lichrosorb- $\text{NH}_2$  column (1).

Further work will be carried out to obtain resolution of this coeluting reaction product.

#### REFERENCE

(1) Murray-M., PME Monthly Progress Report, January 1982, p. 3.

*Michael Murray*

MUM/jig/March 2, 1982

0000143824



PROJECT TITLE : ANALYTICAL INVESTIGATIONS  
PERIOD COVERED : JANUARY 18 - FEBRUARY 19, 1982  
WRITTEN BY : Murray-M. (MUM), Lecoultre-E. (ETL),  
Piadé-J.-J. (JJP), Moser-F. (FMO),  
Genoud-Y. (YVG).

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#### SERVICES FOR OTHER GROUPS

- Upon request from QC, Freon was analyzed in 4 brands of our competitors (FMO).
- The SHI index of 8 experimental cigarettes of project LEAR and of 141 TLA cigarette samples was determined (MUM).
- DMN/NPY and NNN/NNK/NAtB were determined in the mainstream and sidestream smoke of 2 experimental cigarettes of project PROTAGORAS (MUM) (1).
- Trace metals K, Na, Fe, Zn, Mn, Pb, Co, Cu and Mo were analysed in six tobacco extract samples submitted by Biotechnology (MUM) (2).
- Phosphate (6), phosphate/sulfate (13) and alcohol (27) analyses were performed upon request from Product Development and Biotechnology (ETL).
- Headspace analyses were carried out on reference and rejected tipping paper samples upon request from QC (FMO) (3). Additional peaks were found in the chromatogram of rejected sample. Results were confirmed by extraction/GC<sup>2</sup> (YVG).
- Five triacetin samples were analysed for QC by GC<sup>2</sup> (4) (5).
- Free and protein-bound amino acids were analysed in six tobacco extracts provided by Biotechnology and in five RL samples of project TASK (JJP) (6).
- Volatile fatty acids (acetic, propanoic, butanoic) were analysed in 17 RL extract samples by GC (JJP).
- CO and NO mainstream and sidestream smoke deliveries of nine experimental cigarettes of projects SAVOURY and PROTAGORAS were determined (JJP) (7) (8).
- Two fused silica Superox 0.1 coated capillary columns were prepared on request (YVG).

## METHOD DEVELOPMENT

### Polar fused silica capillary columns (YVG) :

A 22 m x 0.3 mm ID fused silica capillary column was coated with Superox 0.1 by the static method. Tests showed an excellent separation efficiency and improved thermal stability over normal Carbowax equivalents. Additional tests were run with tobacco extracts and smoke.

In addition a new technique is presently being investigated to obtain more thermally stable and unextractable films of polar phases on fused silica capillary columns. The technique investigated involves in-situ polymerization of silicon phases by peroxides (9) (10).

### Fermentation monitoring with HPLC (JJJ) :

An easy and rapid method for monitoring the tobacco fermentation products ethanol, methanol, acetic, propanoic, butanoic and lactic acid, glucose, fructose as well as sorbic acid by HPLC is presently being worked out.

## SERVICE FOR PROJECT TASK

After discussion with Product Development further organoleptic examination was made of 17 RL samples. Repeated quantitative analysis of these samples for 12 carboxylic acids indicated a positive correlation between propanoic, phenyl acetic and total acid concentration and undesirable organoleptic properties (Figures 1, 2 and 3) (11) (12).

No correlation was found for lactic acid (Fig. 4) and the other acids which have so far been analysed. No apparent correlation was found between amino acid concentration and negative taste properties (MUM, ETU, JJJ).

## REFERENCES

- (1) Memo from Murray-M. to Schulthess-D., February 17, 1982.
- (2) Memo from Murray-M. to Berney-J., February 11, 1982.
- (3) Memo from Fink-W. to Balliger-P., February 10, 1982.
- (4) Memo from Schwarb-A. to Genoud-Y., January 24, 1982.
- (5) Memo from Genoud-Y. to Schwarb-A., February 24, 1982.
- (6) Memo from Piadé-J. to Berney-J., February 17, 1982.
- (7) Memo from Piadé-J. to Ghiste-P., February 1, 1982.
- (8) Memo from Piadé-J. to Bindler-G., February 19, 1982.
- (9) Sandra-P. et al, HRC and CC 4 (1981) 411.
- (10) Blomberg-L. et al, HRC and CC 4 (1981) 578.
- (11) PME Monthly Progress Report, January 1982, p. 7.
- (12) Memo from Murray-M. to Nyffeler-U., March 4, 1982.

WAR/jig/MARCH 2, 1982 - 7 -

*B. Li*

0000143826

# NEGATIVE TASTE

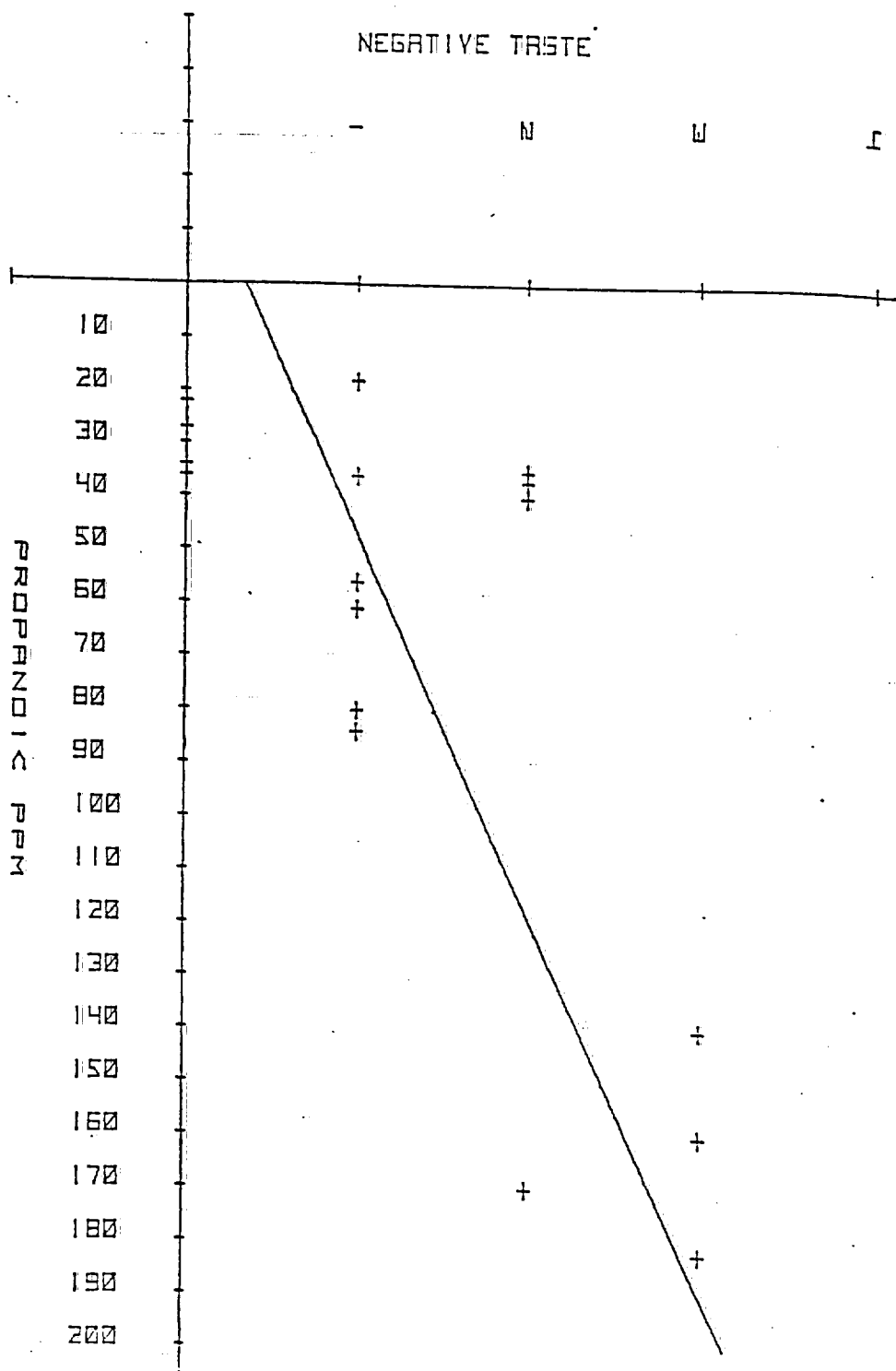
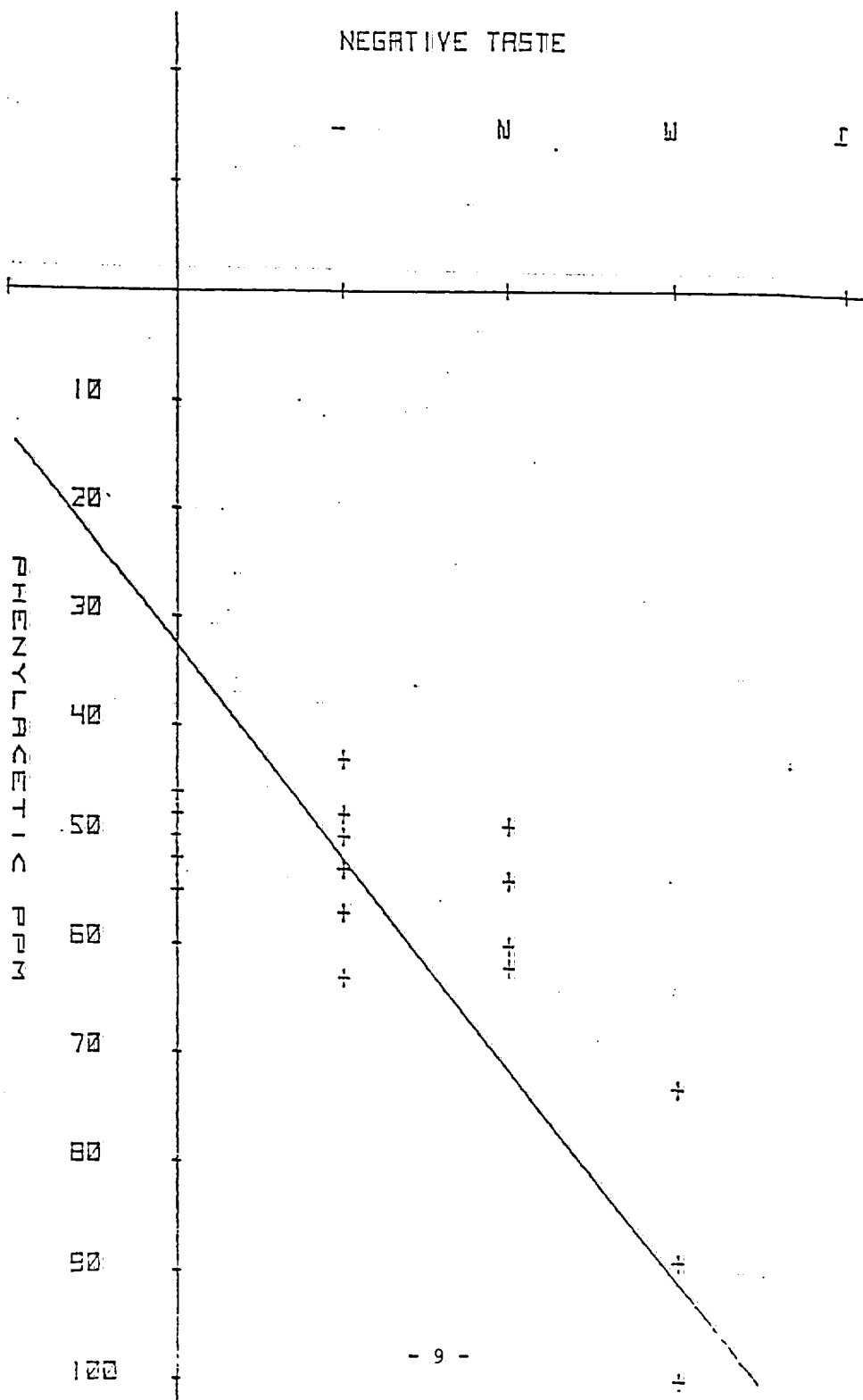


Fig. 1

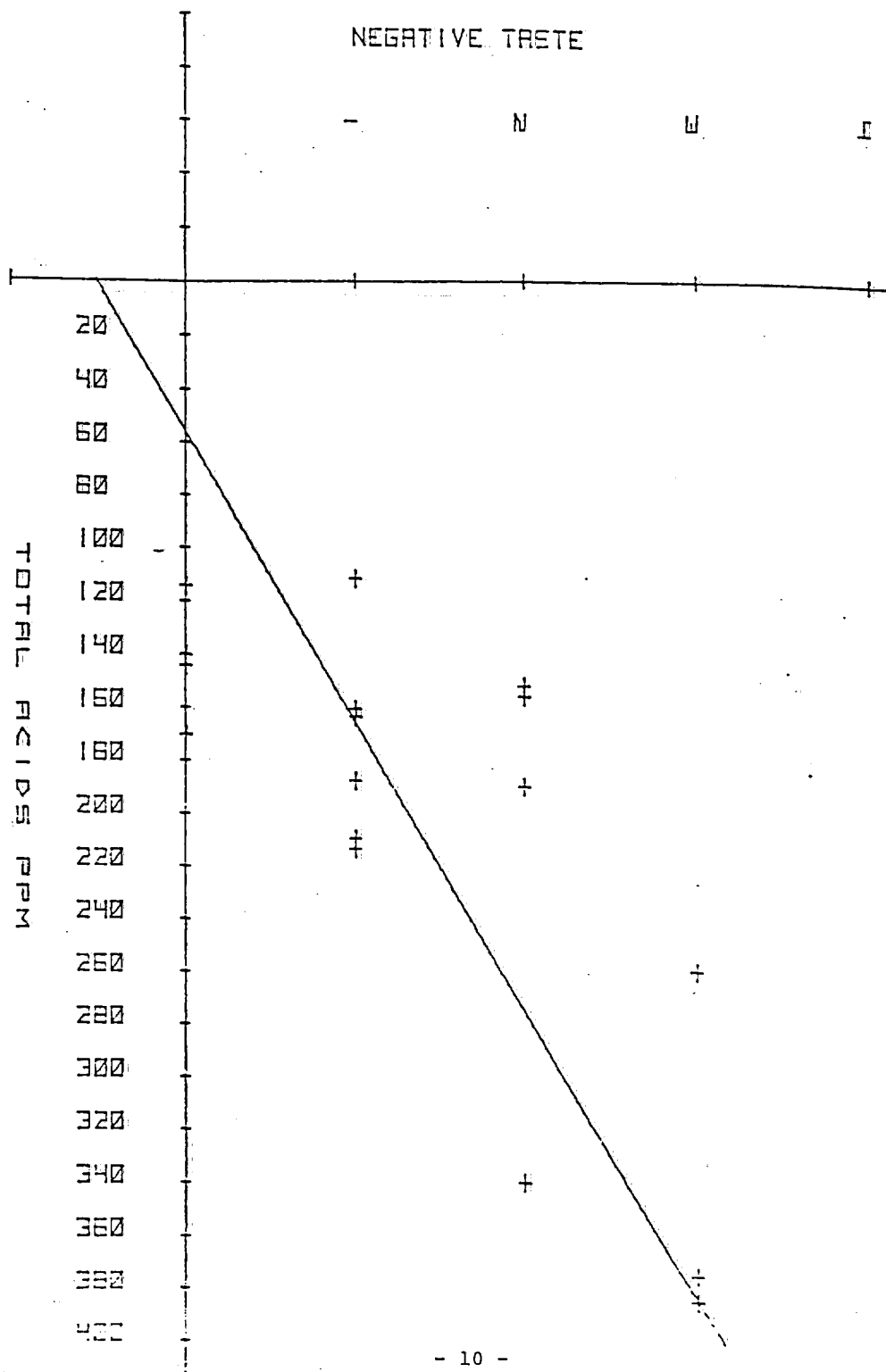
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# NEGATIVE TASTE



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# NEGATIVE TASTE



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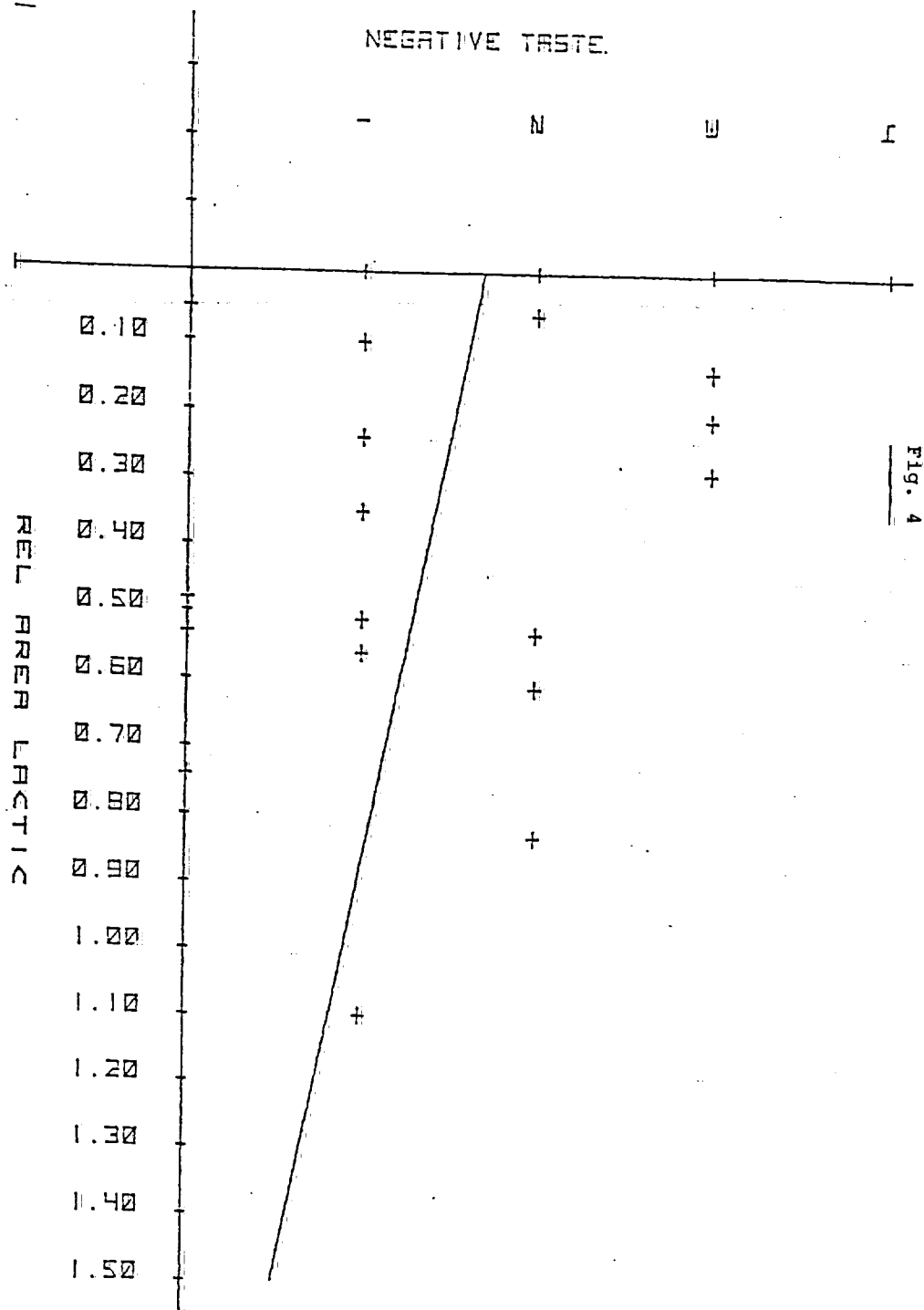


Fig. 4

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PROJECT TITLE : NITRATE-REDUCTION BY CONTROLLED FERMENTATION  
PERIOD COVERED : JANUARY 4 - FEBRUARY 24 1982  
WRITTEN BY : Ruf-C. (CLR)

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## 1. TRIALS

### LEAR Trial 14

After the fermentor had been completely overhauled by a Chemap engineer (1), it was possible to re-start the pilot-plant with LEAR trial 14 on February 1.

The objective of this trial was to perform a complete denitration of the extract and to keep the fermentor sterile over a period of several weeks.

In fact this was achieved for several days at pH 4.5. As soon as the pH was increased, contamination was observed in the fermentor. It had to be stopped after two days and re-inoculated. Because the new centrifuge was not ready for operation, the trial was not run in the closed-loop system during the first two weeks. After the separator had been started-up, the denitrated extract was recycled back to the extractor. After three days of running in this mode of operation, the fermentor was found to be heavily contaminated. Since the pH had been increased just before recycling the extract, it has not yet been determined whether the main cause of contamination was this increase in pH or the recycling itself.

Contamination was also noticed in the extract entering the fermentor despite the fact that the temperature of the sterilization heat-exchangers had been increased and the raw water had been ultra-filtered.

## 2. LABORATORY

In order to have more efficient control of the sterility, some new tests are being introduced by Mr. Hofer in the laboratory programme.

## 3. PRE-ENGINEERING STUDY

The pre-engineering study initiated in October 1981 (2) was completed and distributed. The capital investment and operating costs of three different variants of process plants are presented (3).

4. REFERENCES

1. Lüthi-N, Monthly Report : Pilot-Plant Operations, January 21, 1982
2. Schulthess-D, Monthly Report : Nitrate-Reduction by Controlled Fermentation, October 22, 1981
3. Ruf-C, Lear Project : Pre-engineering Study, February 1982.

*Ruf*

CLR/sde/FEBRUARY 26 1982



PROJECT TITLE : PROJECT LEAR, CIGARETTE PROTOTYPES  
PERIOD COVERED : JULY 23 1981 - FEBRUARY 19 1982  
WRITTEN BY : Borgognon-D. (DIB)

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During the period of the report, three cigarettes were made : one trial and two control cigarettes, bringing the total of LEAR prototypes to 24.

1. TRIAL CIGARETTE

1.1. LEAR 5-17-3

This cigarette was produced with 100% extracted and fermented Burley. It was not necessary to re-apply the fermented extract on the tobacco because the extract was recirculated in a closed-loop system (1).

2. CONTROL CIGARETTES

2.1. LEAR O-17-8

Like cigarette O-17-3, this prototype was made of 100% untreated Burley. This time the tobacco was cut on a KTF production machine in the FTR primary.  
The first time the tobacco was cut on a lab-cutter which led to loose ends in the cigarettes.

2.2. LEAR 6-17-3

This cigarette was the control cigarette for LEAR 5-17-3. It was produced with 100% extracted Burley. The unfermented extract was recirculated in a closed-loop system (1).

3. ANALYSES

The analyses of cigarette 5-17-3 have been received. For cigarettes O-17-8 and 6-17-3, the analyses are under way.

4. REFERENCES

1. Borgognon-D, Notebook 81.06.81, p 2 and 7

DIB/sde/FEBRUARY 26 1982

*S. Burg*

PROJECT TITLE : PILOT-PLANT OPERATIONS  
PERIOD COVERED : JANUARY 20 - FEBRUARY 23 1982  
WRITTEN BY : Lüthi-N. (NIL)

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## 1. EQUIPMENT

### 1.1. Rotocell Extractor

The extraction temperature of 80°C can now be maintained in the extractor by means of the two heat-exchangers which have been installed (1). Since their installation, the occasional presence of nitrite has been able to be avoided (2).

In addition, the level of the extract in the different pumping sections is now kept at a minimum to avoid any possible accumulation of micro-organisms in the extract.

The reduction in this level could be achieved by modifying the extraction system. This modification involved changing the cross counter-current extraction in such a way as to make it work more like that of a simple counter-current.

Also the outlet of the extract is now maintained at a minimum level by means of an automatic level controller.

### 1.2. 20-1 Fermentor

Our small fermentor was given to Chemap for complete servicing.

At the beginning of March, it will again be installed in the pilot-plant and will be ready for further trials.

### 1.3. Sterilizing Filtration

Due to the presence of micro-organisms in the demineralized water in the pilot-plant, a sterilizing filter was installed on the pipe at the position just before the water enters the storage tank of the pilot-plant.

## 2. DRYING OF STRIPS

On February 19, the under-signed went to Quester in Cologne to inspect the purchased drying chamber.

The apparatus was found to be nearly completed and its transport to Neuchâtel is planned for the beginning of March.

3. REFERENCES

1. Lüthi-N, Monthly Report, Pilot-Plant Operations, January 1982 )
2. Lüthi-N, Monthly Report, Pilot-Plant Operations, June 1981

U. All

NIL/sde/FEBRUARY 26 1982

PROJECT TITLE : CIGARETTE AND SMOKE ANALYSIS  
PERIOD COVERED : JANUARY 25 - FEBRUARY 24 1982  
WRITTEN BY : Senehi-F (SEF)

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#### EXPANDED TOBACCO USED FOR THE FIRST TIME IN BELGIAN BRANDS

The analyses showed that the ET material was produced using the Reynolds' process (1).

<u>Manufacturer</u>	<u>Brands</u>	<u>% Expanded Tobacco</u>
Tabacofina	Belga Rouge 70/F	6
Landewyck	Kent 80/F	8
Landewyck	Kent Golden Lights 84/F	15

#### TRANSFORMATION OF US TAR AND SN VALUES INTO GERMAN VALUES

Based on the regression curves recently established for transforming US tar and SN values into ISO values and QA PME tar and SN values into German values (2), two regression curves were calculated for transforming US values into German values (3).

These regression curves are of the type

$$y = ax + b$$

x being the tar or SN obtained by PM Richmond (FTC-US)

y being the tar or SN obtained by PMG (DIN)

For tar and for the same butt length  $y = 1.03 x - 0.9$

For SN and for the same butt length  $y = 1.18 x - 0.21$

For a difference of + 1 mm in butt length found by PMG, the y results (tar and SN) must then be multiplied by 0.98.

These calculations can be made only for cigarettes in the range of

US tar 10 to 18 mg

US SN 0.7 to 1.2 mg

#### QUALITY LEVEL OF PM AND COMPETITORS' BRANDS

Two sampling plans were established for the German and Belgian markets in order to compare the quality level of Philip Morris brands with those of our competitors during a six-month period (4).

DATA FROM THE OFFICIAL FRENCH LABORATORY (LNE) (SEE APPENDIX 1)

A list has been established showing the tar and nicotine values of all PM brands analysed by LNE, compared with the results obtained by QA PME (5).

- All the brands have been declared to correspond to the regulations of article 3 of the decree dated January 30, 1978.
- On average, the tar and SN results obtained by LNE are slightly lower than those obtained by QA PME. For the tar, the 1981 deviation is higher than the 1979 and 1980 deviations.

	<u>Deviation in %</u>		
	<u>1981</u>	<u>1980</u>	<u>1979</u>
Tar	- 6.0	- 3.2	- 3.3
Nicotine	- 1.9	- 3.6	- 3.4

COMPARATIVE RESULTS - FINLAND (SEE APPENDIX 2)

A list has been established showing the DPM, SN and CO values found by the three laboratories

Amer Tupakka OY (ATO)  
Finnish State Laboratory (VTT)  
QA PME

on PM brands sold in Finland (6).

Based on the results obtained by ATO and VTT in 1981 and QA PME results taken from the CIR for the same period, the following comments can be made:

1. Due to the use of the Borgwaldt smoking machine with electrostatic trap, ATO found on average lower smoke yields than VTT and QA PME.

	<u>Deviation in % (VTT-ATO)</u>		
	<u>1981</u>	<u>1979</u>	<u>1978</u>
DPM	- 13.3	- 14.4	- 10.2
SN	- 3.8	- 4.4	+ 2.2
CO	- 2.7	+ 3.8	+ 5.6

	<u>Deviation in % (QA PME-ATO)</u>			
	<u>1981</u>	<u>1979</u>	<u>1979</u>	<u>1978</u>
DPM	- 22.4	- 19.6	- 16.4	- 11.9
SN	- 12.6	- 13.9	- 5.8	- 7.1
CO	- 15.4	- 11.0	- 9.5	+ 8.6

2. Compared with QA PME, VTT who uses identical equipment, found on average lower results in smoke yield.

	<u>Deviation in %</u>			
	<u>1981</u>	<u>1980/1</u>	<u>1979</u>	<u>1978</u>
DPM	- 10.5	- 8.5	- 6.1	- 1.9
SN	- 9.2	- 10.8	- 9.9	- 9.1
CO	- 13.1	- 15.4	- 14.2	+ 2.9

We observed a gradual increase in the DPM deviations, whereas the smoke nicotine deviations were practically constant.

A collective test will be organized to confirm these deviations.

#### SPOTTING COMPLAINTS IN TUNISIA ON MARLBORO PRODUCED IN RICHMOND USA

Following consumer complaints 23 mio of cigarettes from 3 different orders, but from the same shipment, were blocked in a Tunis warehouse. At PM-USA request we carried out an onsite inspection. 72 cases from the three orders were examined.

The percentage of cases having more than 70 % of the cigarettes with critical spotting were estimated as follows:

Order 9081 : about 7 %  
Order 9122 : about 23 %  
Order 9123 : about 25 %

A complete report will be issued soon.

#### PRODUCT REPORTS

Product reports were written on the following new or modified brands:

<u>Brand</u>	<u>Manufacturer</u>	<u>Country of sale</u>
Parisienne Extra 79/F (line extension)	Burrus	Switzerland
Peter Stuyvesant Ultra 84/F (line extension)	Laurens- Rothmans	Switzerland
Craven A Luxury Length 94/F (line extension)	Carreras- Rothmans	United Kingdom
Craven A Luxury Length Special Mild 94/F (line extension)	Carreras- Rothmans	United Kingdom
Silk Cut Extra Mild 84/F (national relaunch)	Gallaher	United Kingdom

REFERENCES

- 1 Letter from Senehi-F. (February 17 1982)
- 2 Monthly report October 23 - November 23 1981
- 3 Telecopy from Senehi-F. to Völkl-G (February 23 1982)
- 4 Letter from Senehi-F. to Lutzig-B.W. and Fauville-H.  
(January 18 1982 / February 22 1982)
- 5 Letter from Senehi-F. (February 23 1982)
- 6 Letter from Senehi-F. (February 16 1982)

*Senehi*

Enclosures: Appendix 1 and 2

SEF/edk/FEBRUARY 25 1982

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COMPARATIVE RESULTS BETWEEN THE  
OFFICIAL FRENCH LABORATORY (LNE) AND QA PME

APPENDIX 1

Brand	Values printed on pack		Limit Values		LNE		QA PME		CIR (10-11/1981)	
	Goudrons	Nicotine	Goudrons	Nicotine	Goudrons	Nicotine	Goudrons	Nicotine	Goudrons	Nicotine
AMADA MENTHOL	17.9	1.09	20.6	1.25	15.8	1.14	17.9	1.17	16.7	1.15
AMADA GALION 100'S	17.9	1.09	20.6	1.25	16.8	1.07	17.5	1.10	16.7	1.15
HESTERFIELD F. SOFT	15.0	1.20	17.3	1.38	13.9	1.14	14.6	1.13	15.0	1.21
HESTERFIELD K.S. NF	21.0	1.60	24.2	1.84	20.8	1.69	22.5	1.69	21.8	1.74
HESTERFIELD REGULAR	21.0	1.49	24.2	1.71	19.0	1.47	20.1	1.48	19.0	1.46
G M FILTER KING	15.0	1.20	17.3	1.38	14.2	1.23	15.0	1.19	14.6	1.19
ARK F. KING SIZE	17.8	1.34	20.5	1.54	15.2	1.23	15.5	1.23	15.9	1.33
WARLBORO BOX	15.8	1.09	18.2	1.25	15.6	1.16	16.9	1.22	16.8	1.18
WARLBORO K.S. SOFT	15.8	1.09	18.2	1.25	16.1	1.24	16.9	1.21	16.3	1.15
WARLBORO MENTHOL	15.8	1.09	18.2	1.25	15.2	1.15	16.7	1.18	16.8	1.12
WARLBORO 100'S	16.9	1.20	19.4	1.38	15.6	1.24	16.7	1.33	16.8	1.27
HERIT	7.5	0.56	8.6	0.64	7.3	0.57	8.0	0.55	8.0	0.54
MURATTI AMBASSADOR BOX	12.2	0.92	14.0	1.06	12.0	0.77	12.3	0.80	12.0	0.80
MURATTI AMB. EXTRA MILD	6.7	0.45	7.7	0.52	6.9	0.42	7.1	0.41	7.0	0.43
PHILIP MORRIS FILTER	15.8	1.09	18.2	1.25	15.6	1.13	16.7	1.20	15.9	1.16
PHILIP MORRIS INT.	15.9	1.09	18.3	1.25	15.1	0.95	15.4	0.98	16.0	1.02
PHILIP MORRIS SUP. LIGHT	3.9	0.40	4.5	0.46	3.5	0.32	4.1	0.36	4.0	0.35
MULTIFILTER 100'S	13.9	0.98	16.0	1.13	12.2	0.80	12.9	0.86	12.0	0.87
GENERAL AVERAGE	14.77	1.05			13.93	1.04	14.82	1.06	14.52	1.06

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Brands	DPM (mg/cig)			Smoke nicotine (mg/cig)			Carbon monoxide (mg/cig)		
	ATO	VTT	QAPME	ATO	VTT	QAPME	ATO	VTT	QAPME
<u>1981/1st test</u>									
MLF 03	14.1	15.9	17.0	0.96	1.00	1.06	14.5	13.9	16.0
MFM 01	14.5	17.1	17.2	0.98	1.02	1.05	13.9	15.0	16.0
MLL 03	9.3	10.2	12.7	0.63	0.71	0.79	8.3	9.0	11.1
PMM 01	13.2*	13.7*	17.1*	0.92	0.89	1.04	14.9*	14.7*	19.4*
BEM 01	9.2	11.9	11.9	0.68	0.71	0.80	9.8	10.4	11.3
BEN 01	7.4	8.8	10.8	0.52	0.57	0.74	8.1	9.2	12.7
BEO 01	7.4	8.6	10.5	0.53	0.56	0.70	7.9	8.6	9.6
Average $\bar{x}$	10.3	12.1	13.4	0.75	0.78	0.88	10.4	11.0	12.8
<u>1981/2nd test</u>									
MLF 03	14.8	16.6	17.4	1.01	1.03	1.06	14.5	14.7	15.8
MFM 01	13.9	16.0	17.4	1.00	1.04	1.03	14.5	14.4	16.0
MLL 03	9.7	10.9	12.8	0.70	0.70	0.78	9.1	9.3	10.8
PMM 01	13.1*	14.6*	----	0.87*	0.89*	----	15.1*	14.7*	----
BEM 01	9.4	10.3	12.4	0.74	0.74	0.87	8.6*	8.8*	----
BEN 01	7.4	9.4	10.8	0.56	0.62	0.74	8.2*	9.1*	----
BEO 01	7.1	8.6	10.4	0.56	0.62	0.69	9.4	8.9	10.4
Average $\bar{x}$	10.4	12.0	13.5	0.76	0.79	0.86	11.9	11.8	13.3
General average for 1981 $\bar{x}$	10.4	12.0	13.4	0.76	0.79	0.87	11.0	11.3	13.0

\* This result is not taken into consideration for the average

17.2.82 SE edk

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PROJECT TITLE : QA ANALYTICAL SERVICES  
PERIOD COVERED : JANUARY 26 - FEBRUARY 25 1982  
WRITTEN BY : Widmer-A (ALW)

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#### 1.5. LEGISLATION

- Denaturation of rum and ethanol intended for UK cigarettes.  
The question was raised by PMG-Munich as to which denaturation agents are allowed to be used in the UK (1).  
The problem was examined by "R+D Technical Services" (2):
  - According to list 3 of the Hunter list:  
Ethanol and rum, which may be used as additives in tobacco products, must be denaturated with not more than 0.05 % of nicotine salts (calculated as nicotine).
  - According to list 2 of the Hunter list:  
Butane-2-one (methyl ethyl ketone) is mentioned in the list of products which may be applied to the tobacco blend. Their total level must not exceed 0.15 % of the tobacco blend in cigarettes, calculated on a dry weight basis.

Butane-2-one and ethanol have the same boiling point and are evaporated together during tobacco processing. The residual amount of the former will not significantly increase the total amount of additives put in the blend which are mentioned in list 2. As Butane-2-one is already used as a denaturation agent for ethanol in Germany, we recommend its use for the preparation of AC-solutions for UK cigarettes (3).

#### 2.1. CASING KITCHEN

- MAG-AC  
The preparation procedure was established for this new solution (4).
- PZ-8-FI  
The preparation procedure had to be modified following the acquisition of a new recipient (5).

#### 3.2.1. TRIALS WITH TOBACCO INGREDIENTS

- Invert sugar (M. BORGWALDT, Hamburg; "GLYCARMEL 4952" / yellow sheet 6189, 100 kg)  
This product is used as a standard material in PMG.  
An industrial trial (MLF-CH) was organized in order to qualify this product for FTR.

Laboratory: The sample is within specifications.  
Smoke analyses: No significant difference between the standard and the trial was found.  
Subjective evaluation (Panel B, discriminative test):  
A significant difference (99 to 99.5 %) between the trial and the standard was found.  
Furthermore, as the drum did not have the usual dimension the product could not be liquified on the installation in the casing kitchen.

After discussions with the supplier concerning the dimensions of the drums, the trial might be repeated.

- Liquid sugar

Liquid sugar instead of raw cane sugar is used by PMH for the preparation of BURLEY CASING. For handling and price reasons, the possibility of applying liquid sugar in PMG-Berlin and Munich was studied.  
Four reference samples of different shipments to PMH were analysed:

	Fructose+Glucose (HPLC) (%)	Saccharose (HPLC) (%)	Water (KF) (%)
1	10.5	56.8	33.3
2	10.3	50.9	36.8
3	8.8	56.9	32.5
4	22.4	43.7	33.1

Furthermore the influence of the storage time was studied on a fresh sample:

	Fructose+Glucose (HPLC) (%)	Saccharose (HPLC) (%)
Upon delivery	12.8	57.0
After two months	19.5	50.2

The above-mentioned results show a certain doubt about the regularity of the shipments as regards the composition of different sugars. Furthermore, depending on the storage time and conditions, a decomposition of saccharose into fructose and glucose seems to take place.

For this reason, and also because commercially-available samples from different suppliers show considerable differences in the composition of the sugars, their influence on the cigarette taste was evaluated.

Test-cigarettes (MLF-IT) were produced in PMG-Munich with the following test materials:

	Fructose + Glucose (HPLC) (%)	Saccharose (HPLC) (%)	Water (KF) (%)
- Invert sugar SUCRERIE TIRLEMONT (BE)	77.6	< 0.5	20.2
- Liquid sugar SÜDZUCKER (GER)	- 2.3 *	65.6 64.2 *	33.2
- Liquid sugar COMMERCIALE SUCRERIE (FR)	- 22.8 *	65.9 44.5 *	32.9
- Liquid sugar PFEIFFER&LANGEN (GER)	41.4 45 *	27.5 23 *	31.1

\* indication of the supplier

The total amount of sugar was identical in all the trials; it was calculated on the basis of the standard recipe of BURLEY CASING.

Smoke and tobacco analyses: No significant difference between the standard and the trials was found except in the contents of reducing sugars which reflect the quantity of the additions.

Subjective evaluation (Panel A): A significant difference between the standard and the trials was found. None of the trials were accepted (6).

A further trial (MLK-21) with the standard material used in PMH (supplier: SUIKER UNIE) was carried out by PMG-Berlin.

Laboratory: The composition of the sample was found to be as follows:

Fructose+Glucose (HPLC)	(%)	12.4
Saccharose (HPLC)	(%)	54.4
Water (KF)	(%)	33.0

Smoke and tobacco analyses: No significant difference between the standard and the trial was found except in the contents of reducing sugars.

Subjective evaluation (Panel A): A significant difference between the standard and the trial was found. The trial was refused (7).

A further repetition of this trial in FTR, including a panel D test, is planned.

### 3.2.2. QUALITY CONTROL OF TOBACCO INGREDIENTS

- Honey (J. SCHUETTE & CO, Bremen (GER) / blue sheet 9084)

Two drums out of 33 had to be refused. Reason: In one drum an accumulation of solid particles (impurities) was found on the surface. The contents of the other drums had a strongly pronounced acid taste. This was probably brought about by the fermentation which was then stopped by heat treatment.

### 3.4.1. TRIALS ON FILTER COMPONENTS

- Activated charcoal (CHEMVIRON, Zürich; "UKCT"/yellow sheet 5418, 14.4 t and "MF III" / yellow sheet 5428, 14.4 t)

These materials were tested on their own or blended with the standard charcoal from PICA. The results were negative (8). The Purchasing Department proposed blending the above-mentioned quality with charcoal from PICA. In this case the former made up 5 % of the blend. This operation would take place in the production center in Levallois (FR) (9). However, the installation in Onnens, producing the mixture "CAPO 46", has a free capacity. Blending one quality of CHEMVIRON charcoal in a 1:10 ratio to PICA charcoal would occupy the installation for 93 days (10).

It was decided to run a trial with 10 % charcoal from CHEMVIRON blended with 90 % charcoal from PICA. Reason: The installation in Onnens guarantees a homogeneous blend and the operations would be supervised by QA.

#### "UKCT":

Laboratory: The results of the blend are within specification.  
Smoke analyses: No significant difference between the trial and the standard was found.

#### Subjective evaluation:

- Panel B (discriminative test): No significant difference between the trial and the standard was found.
- Panel D : 193 answers were taken into consideration for statistical evaluation. No significant difference was stated between the trial and the standard (11).
- Panel A : A significant difference between the trial and the standard was found. The trial was refused.

#### "MF III":

Laboratory: The results of the blend are within specifications.

Smoke analyses: No significant difference between the trial and the standard was found.

#### Subjective evaluation:

- Panel B (discriminative test): No significant difference between the trial and the standard was found.
- Panel D : 216 answers were taken into consideration for statistical evaluation. No significant difference was stated between the trial and the standard (12).
- Panel A : A significant difference between the trial and the standard was found. The trial was refused.

- 26 -

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### 3.7.1. TRIALS WITH CIGARETTE SEAM GLUES

- "SICHOCOLL STN 5033" (HENKEL AG, Düsseldorf (GER))

This type is a starch-based paste. PMG-Munich evaluated this quality on MLF-IT cigarettes with a view to finding an alternative to its own production.

Machineability: Better than the standard glue.

Smoke analyses: No significant difference between the standard and the trial was found.

Subjective evaluation (Panel A): No significant difference between the standard and the trial was found. The trial was accepted.

On the basis of these results it was decided that this type can be used as a standard material (13).

### 3.7.2. QUALITY CONTROL OF CIGARETTE SEAM GLUES

- "LESSO 1487 X-3" (LAESSER AG, Erlinsbach)

This is the standard starch-based liquid glue which is applied through a KAYMICH-nozzle. The producer guarantees the quality of this liquid glue for three months as from its day of production.

In the beginning of December, simultaneously in the production centers of Serrières and Cousset, the glue was found to be too liquid. Even by applying it at room temperature and without pressure, serious glueing problems were encountered. An inspection of all the drums stored in Serrières, Cousset and Onnens showed that part of the glue had started to ferment. Fermentation causes a rapid decrease in pH and viscosity. One of the most affected drums showed for example a pH of 5.4 instead of > 7.4 and a viscosity of 1600 cps instead of > 14900 cps.

All the affected drums were blocked immediately and replaced by new ones. During the period of December 81 and January 82 a beginning of fermentation was detected during the reinforced control on further shipments of this glue produced at different dates. 25 out of 65 drums had to be refused.

Up till now, the producer has not been able to give any explanation of this decrease in quality.

The following measures were taken:

- The glue is produced every Friday and supplied every Tuesday. The quantity ordered by FTR just covers our needs from one supply-day to the next. This means that the glue has been entirely used up 12 days after its production.
- The changes in the glue application systems planned for Serrières and PMG-Munich have been suspended until the quality problems of the glue have been resolved.

### 3.14.2. QUALITY CONTROL OF ETNA

- A new manual for Quality Control was established (14).  
The following main modifications were introduced into the new manual (15):
  - All analyses on the cut rag are done by the Production Control in Serrières.
  - Chemical analyses are only done on expanded tobacco. A reference sample of cut rag will be retained.
  - All physical analyses on the expanded tobacco after storage have been abandoned, except for the determination of the filling power.

### 3.15.2. QUALITY CONTROL OF RCB

- A new manual for Quality Control was established (16).  
The following main modification was introduced in this new edition (17):
  - Weekly chemical analyses are done on the final product. However, regular TL-analyses will only be done every four months, instead of every month.

### 3.16.2. QUALITY CONTROL OF ESTHER


- A manual for Quality Control was established (18).  
The following main modification, compared with the routine procedure, was introduced:
  - Chemical analyses on the final product are performed on every fifth batch per blend.

### 5.3. ASSISTANCE TO OTHER AFFILIATES

- Analyses for the ETNA-plant in PMH-Bergen op Zoom  
Humectants in tobacco (18 samples)
- Analyses for the ETNA-plant in PMG-Munich  
Humectants in tobacco (2 samples)

# 7

## REFERENCES

- 1 Letter from Janke-W to Lopes-F (January 11 1982)
  - 2 Letter from Keagy-K.B. to Widmer-A (February 5 1982)
  - 3 Letter from Widmer-A to Janke-W (February 15 1982)
  - 4 Letter from Schwarb-A to the CASING KITCHEN (February 3 1982)
  - 5 Letter from Schwarb-A to the CASING KITCHEN (February 8 1982)
  - 6 Letter from Widmer-A to Lutzig-B.W. (March 20 1981)
  - 7 Letter from Widmer-A to Tessendorf-W (February 15 1982)
  - 8 Monthly report Widmer-A (February 1981)
  - 9 MPP & QA Coordination Meeting (April 4 1981)
  - 10 Letter from Caccivio-J.P. to Schwarb-A (October 14 1981)
  - 11 Letter from Stampfli-M to Widmer-A (December 1 1981)
  - 12 Letter from Stampfli-M to Widmer-A (February 4 1982)
  - 13 Letter from Widmer-A to Lutzig-B.W. (February 15 1982)
  - 14 "ETNA/procédé contrôle qualité" from Widmer-A and Matthey-A (February 15 1982)
  - 15 "ETNA/procédé contrôle qualité" from Widmer-A (January 8 1981)
  - 16 "RCB / procédé contrôle qualité" from Widmer-A and Matthey-A (February 15 1982)
  - 17 "Cahier des charges, MONIQUE" from Joseph-L (May 1979)
  - 18 "ESTHER / procédé contrôle qualité" from Widmer-A and Matthey-A (February 15 1982)
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ALW/edk/MARCH 8 1982

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PROJECT TITLE : SPECIFICATIONS  
PERIOD COVERED : 26 JANUARY - 25 FEBRUARY 1982  
WRITTEN BY : Kopp-A.-M.

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## 1. FTR-Switzerland

1.1. Tobacco cutting was standardized to the PM USA width of cut and after trials was brought up from 0.80 to 0.85 mm for all the blends except :

- Etna (0.80 mm)
- Maryland blends (0.70 mm)
- PMI blend (0.70 mm).

## 1.2. Changes in Processing

- New blend MA 006 for MURATTI AMBASSADOR EXTRA MAG. The existing blend MA 001 remains valid for MAB and MAH.
- Some processing specifications have been brought up-to-date following the modification in the rate of addition of shorts in the different groups of blends.

## 1.3. Changes in Cigarettes

- Cigarette MAG 01 has been replaced by MAG 02 (project COLORADO) with a new blend, new tobacco weight, new tipping paper and cigarette paper. The filter supplied by Baumgartner remains the same.

## 1.4. Changes in Packing

- The vend' pack version MAG 003 has been withdrawn. MAG 001 sold in Switzerland and 3's pack MAG 002 have been updated.
- Several specifications have been brought up-to-date, mostly due to price increases of Swiss brands beginning in May 1982.

1.5. A complete file of project specifications has been issued for project "GAMMA" - PHILIP MORRIS SUPER LIGHTS PMS 03 for sale in Norway.

## 2. PM-Holland

### 2.1. Changes in Processing

- As Esther is again being used in Marlboro blend ML 017, 31 liters instead of 29 liters of MF-AC is used.

## 2.2. Changes in Packing

- New specification for PHILIP MORRIS FILTER KINGS PMB 090 for sale in Holland.

2.3. The first edition of PHILIP MORRIS FILTER KINGS PMB 01 has been produced. It corresponds to PFF 02 with a new logotype (project JOHNNY II).

2.4. A complete new file for the second version of TENNIS 53 (MLB-UK) has been issued with a new blend, new solutions and new Burley treatment. The code of this cigarette is now MLB 04.

## 3. PM-Munich/Berlin

3.1. The processing specifications of PM 002 and MG 003 have been brought up-to-date (new amount of Burley).

3.2. The MLF-108 filter specification has been modified (filter paper HF 24K 28 variant 4/2 has been approved as an equivalent to FU-POV 24 L).

3.3. The cigarette specifications of MER 02/03 have been modified following the PME modification request to use the approved modified Z 4/100 in order to reduce the smoke numbers.

3.4. The packing specifications of MLB 040 and MLB 041/49 have been modified as the inner frame Cosmocart 230 g/m<sup>2</sup> is now used instead of Inv. D 240 g/m<sup>2</sup>. This quality has been accepted as inner frame board exclusively and by Marketing DB for all brands sold in Germany.

3.5. A new version of PMS 069 PHILIP MORRIS SUPER LIGHTS in display carton, for XD (KLM) sale has been issued. PMS 064 has been modified for XD sale in display carton.

3.6. A new cigarette code MHC 03 has been given for 3's sample packs MARLBORO 100 - UK which will be produced in Munich with blend ML 028 - MLB-UK, ex PMH.

## 4. PMB-Brussels

4.1. A new cigarette specification for MLF 29 MARLBORO FILTER has been issued using blend ex PMH ML 017 (ES 3.3% and ET 6.0%) for sale in France (MLF 225).

4.2. The packing specifications of MER 054/55 have been updated for the new health warning applied in Belgium.

## 5. Licencees

### 5.1. Greece

The filter-making specifications of MLK 120 and cigarette MLK MARLBORO KING SIZE have been modified following the reduction in filter and cigarette diameters to 7.90 and 7.95 respectively.

### 5.2. Hungary

A new complete file has been issued for MLK 24 MARLBORO KING SIZE made in Eger.

### 5.3. Italy

Following the second step of project ALFA to reduce the smoke numbers, provisional specifications have been issued for MERCEDES KING SIZE made in Florence :

- Processing of ME 003 with modified blend;
- Cigarette-making of MEK 01 (with new standard diameter for filter and cigarette);
- New filter for MEK 120 No 34.6300 issued by Intertaba.

## 6. PME Standard Recipes

- Some recipes have been updated (projects TENNIS - MLB-UK and GAMMA - PMS).
- Reinstatement of MLN-AC No 12.2085 withdrawn in 1979. Use in PMG for mentholisation of blend for shell and slide pack.
- New recipe MLB-UK2-AC No 12.2211 for MLB-UK ex PMH (project TENNIS).

## 7. PME Material Specifications

7.1. The following material specifications were submitted to the suppliers :

- Filtration material 3.0/43'000 I and 3.0/43'000 Y to RHODIACETA;
- Cigarette paper Ivy Verge MCHP 5 (M) to FLETCHER (MER-UK/BE);
- Tipping paper TP 175 to TANN, Acquafuge 944 BNG to MALAUCENE (MAG/CH and PMU/DB) and Acqualiège 66-32 E to MALAUCENE (BSL/DB).

7.2. Material specifications approved by the suppliers :

Benkert and Schoeller & Hoesch have now approved all the material specifications which were submitted.

8. Specifications on EDP

All the files of the AMAPS system except one (Purchasing Control) are working successfully in a test environment, with the five pilot brands entered into the system.

FTR's Purchasing and QA Services have agreed that the Bill of Material and the Revision files will be kept up-to-date by FTR's QA Specifications Office. This was recommended to the QA and Purchasing managers of the region who attended information sessions in Neuchâtel in mid-January and at the end of February respectively.

The Bill of Material file in AMAPS will replace 90% of the present specifications, as regards material and product structure.

Some definitions concerning the Bill of Material and Revision files were outlined by the two company functions involved.

As regards the non-material specifications (sizes, moistures, etc), the development work is being continued. Some thought was also given to the input screens to be prepared, as well as to the links existing between the various sets of information.

A complementary UFO programme is in preparation to resolve the kitchen recipe problem.

*atoh*

PROJECT TITLE : COMPUTER AND INSTRUMENTATION  
SUPPORT GROUP

PERIOD COVERED : 23 JANUARY - 22 FEBRUARY 1982

WRITTEN BY : Thévoz-M.

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Automation of the Smoking Laboratory (Project COLDAC)

The final structure of the data base has now been established. It is based on the information collected from the specification lists of the various users and on numerous simulations made on a demonstration data base. The final version, which we shall install onto the HP 1000 during the first two weeks of March, comprises 256 analytical criteria and will have a capacity of 11000 inputs. Access and querying are carried out via about 10 keys of which the most important are :

- the sample code - cigarette code;
- analysis month - production centre;
- production date - country of sale.

Keys which are more specific have been planned for development tests and certain TLA. A 45-Mbyte free space has been reserved on the disc for storing all the analytical data of the Smoking Laboratory. A 8-Mbyte free zone has been kept for future extensions of the data base.

A detailed analysis of the specification lists shows that it will be necessary to write about 200 programmes in FORTRAN, each having 25 to 60-Kbyte codes. This considerable amount of work results firstly from the requirements of the users (which, it should be mentioned, are totally justifiable) concerning the editing of the stored data and secondly, from the problems posed by data acquisition in real time on laboratory instruments.

We are currently working on the development of the main querying programmes of the data base. A demonstration version has already enabled us to produce a complete routine report with all the calculations from a bar code which is read optically on a label.

The calculations in this report include statistical tests (DIXON) enabling us to delete out of range values. A programme allowing dynamic management of the questions - answers (menu) on the screen of the terminals is being studied by our group.

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Data handling in protected fields by a screen formula offers the possibility of simple dialoging between the operator and the programme to be performed. This approach also affords greater security of operation and enables the validity and nature of the operator's answers to be checked locally.

#### Miscellaneous

The firmness-testing instruments for QC Richmond were sent at the end of January. Other units are in preparation for various PME affiliates and Canada.

#### Training Courses

A 2-day course was given by Hewlett-Packard, Grenoble, on HP 3078 interfaces (Data coupler) and this enabled us to become familiar with certain complex problems concerning the instrument - computer link-up. Regarding this matter we have developed a demonstration programme for automatic reading of Mettler AK 160 scales controlled by the HP 1000.

*huv*

MIT/jud/FEBRUARY 24, 1982

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PROJECT TITLE : DOCUMENTATION AND LIBRARY 1  
PERIOD COVERED : JANUARY 12 - FEBRUARY 19 1982  
WRITTEN BY : Keagy-K-B. (KBK)

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#### Legislation

1. A question concerning the legislation governing the denaturation of ethyl alcohol and its subsequent application to tobacco was submitted. Answers were requested for Switzerland, Great Britain, Italy, Belgium, the Netherlands. The information on France and West Germany and the other members of the EEC is not so urgently required.

From the literature available in our R & D Library it was easy to find the answers for Switzerland and Great Britain, where methylethylketone (or 2-butanone) is permitted as a denaturant of ethyl alcohol and where denatured alcohol may be used on tobacco. (This information is accessible on Stairs). For the other countries, outside sources must be consulted and no answer has been compiled yet.

2. The backlog of documents regarding tobacco legislation in Great Britain has been analyzed and is available on Stairs.

*KB Keagy*

KBK/kbk/FEBRUARY 19 1982

PROJECT TITLE : DOCUMENTATION AND LIBRARY 2  
PERIOD COVERED : Up to 24 FEBRUARY 1982  
WRITTEN BY : Lüdin-R.

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All the documents which are kept in the library or which are on permanent loan to the departments are analysed and put on STAIRS.

By 31 January 1982, 24'300 documents had been entered into STAIRS; they comprise :

- 3'100 books
- 11'620 separata, etc.

Another scientific library with which we have close relations is connected to our data-base in Neuchâtel via the public telephone net-work. This service wishes to enter all its scientific and technical documentation into STAIRS and in January 1982 they put the first 50 documents into the system.

In the near future, the library will establish, on an experimental basis, a connection to an external data base, Data-Star, which is situated in Bern. This data-base comprises about 10 files with chemical, biological, technical, economic etc information and it will complement our own data base.

We plan to enter into STAIRS information on the most important developments in online services, office automation and viodetex systems.

*R. Lüdin*

ROL/jud/FEBRUARY 24, 1982

0000143856



PROJECT TITLE : PATENTS  
PERIOD COVERED : JANUARY 17 - FEBRUARY 16, 1982  
WRITTEN BY : Mandiratta-J-C. (MJA)

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BENKERT'S WEST GERMAN PATENT APPLICATION P 25 31 285  
- ELECTRICAL PERFORATION OF TIPPING PAPER -

During my recent visit to Richmond the prior art was reviewed and it was decided by the Foreign Patent Committee to oppose the above-mentioned application. Instructions to do so were sent to Dr. Hach with the prior art.

NEW APPLICATIONS

The following applications have been filed this month for the under-mentioned West German priorities:

P 31 01 768: Apparatus for extracting soluble constituents from cut plant material.

This case is a European Patent application designating Switzerland, the Netherlands, Belgium, Great Britain, France and Italy.

P 31 05 454: Smoking articles with filters and apparatus for producing the same.

P 31 05 455: Apparatus for smoking channels in the periphery of filter elements.

P 31 05 456: Rod-like article with secondary air channels and apparatus for producing such an article.

These three cases were filed via the European Patent Office in Switzerland, the Netherlands, Belgium, Great Britain, France,

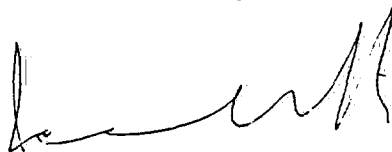
Italy and Sweden; a national application was filed in Finland.

Complete applications and filing documents were sent to the Patent Office in Richmond for filing US applications.

#### FORESEEN FILINGS

Details of the under-mentioned filings were given to Messrs. Bovard & Co., Patent Attorneys in Bern, for preparing European Applications to file in West Germany, Great Britain, Italy and Switzerland:

1. Automatic box filling and palettizing system.
2. Filter trays unloading system and massflow feeding to a cigarette maker.



MJA/mle/February 17, 1982

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